

TITRIMETRIC PROCEDURE FOR ACID-SOLUBLE AND ACID INSOLUBLE SULFIDES
EPA 9034 REVISION O 1996

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Facility Name: _____ VELAP ID _____

Assessor Name: _____ Analyst Name: _____ Inspection Date _____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
If samples were pretreated in the field with aluminum chloride, allowed to settle and flocculate, and the clear supernatant collected for analysis of dissolved sulfide, was this method not used?	2003 NELAC 5.5.7.3- doc. of collection information				
Was potassium iodide solution standardized against either standardized phenylarsine or sodium thiosulfate?	5.6.1				
Was the normality of the potassium iodide calculated by the equation: $I_2N = ((\text{mL of titrant} \times \text{normality of titrant}) / \text{potassium iodide sample size in mL})$	5.6.3				
Were standards prepared at a pH between 9 and 11?	5.7				
Were aqueous samples taken with a minimum of aeration by steps such as filling sample containers so as to avoid headspace and stoppering?	9030 3.1, 6.2				
Were aqueous samples and effluents preserved with 2N zinc acetate at four drops per 100 mL of sample?	9030 6.2				
Were aqueous samples and effluents adjusted to pH > 9 with sodium hydroxide?	9030 6.2				
Did solid samples have their surfaces moistened with 2N zinc acetate?	9030 6.2				
Were samples cooled to 4°C and stored headspace free for not longer than 7 days?	9030 6.2				
Was this method used to quantify the sulfide concentrations of samples distilled by EPA Method 9030?	7.1				
Was a known amount of standardized I ₂ solution added to a container that was known to be in excess so that sulfides could be oxidized?	7.3.1				
When the distillation for acid-soluble sulfide was performed, was 6N HCl added to the iodine?	7.3.2				
Were the gas scrubbing solutions obtained from 9030 pipetted into the container while keeping the pipet tip below the surface of the iodine solution?	7.3.3				

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Were additional known amounts of standardized iodine solution added to the containers if, at any point, the amber color disappeared or faded to yellow?	7.3.3				
Were scrubbing bottles lastly rinsed into containers with reagent water?	7.3.5				
Were solutions in the containers titrated with standard phenylarsine oxide or sodium thiosulfate solution until amber color faded to yellow?	7.3.6				
After solutions in the container faded to yellow, was enough starch indicator added to them to turn the solutions dark blue, and the solutions titrated again until blue disappeared?	7.3.6				
Was the following equation used to calculate sulfide concentration: $((\text{mL I}_2 \times \text{N I}_2) - (\text{mL titrant} \times \text{N titrant})) \times (32.06\text{g}/2 \text{ equivalents}) / (\text{sample weight}(\text{kg}) \text{ or sample volume}(\text{L}))$	7.3.7				
Were reagent blanks analyzed the more frequent of either once in 20 samples or once per analytical batch?	8.2				
Were check standards prepared from known amounts of sulfide in water and run with each analytical batch or once in 20 samples?	8.3				
Were matrix spike samples brought through the whole preparation and analytical process and analyzed the more frequent of either once each analytical batch or once in 20 samples?	8.4				
Notes/Comments:					